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CHEMICAL, PHYSICAL AND MECHANICAL PROPERTIES OF LOW DENSITY PHOSPHATE ESTER HYDRAULIC FLUIDS

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TECHNICAL REPORT AFML-TR-73-78

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FOREWIND

This report was prepared by the Lubricants and Tribology Detach of the Nonmetallic Materials Division, Air Force Materials Laboratory. Air Force Systems Command, Wright-Patterson Air Force Base, Ohio. The work was conducted under Project 7340, "Nonmetallic and Composite Materials", Task No. 734008, "Energy Transfer Fluids". Herbert Schwenker and F. C. Brooks were the Project Engineers.

The purpose and results of the effort expended between March 1970 and June 1972 are presented.

The manuscript was submitted for publication by the autnors in August 1972.

This technical report has been reviewed and is approved.

Kenneth a Kivis

MEMORY A. DATIS. MAJOR, USAF Chief. Lubricants and Tribology

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ABSTRACT

Three low density phosphate ester fluid candidates, MLO-70-32, MLO-70-62 and MLO-71-37, were characterized as to their physical and chemical properties. MLO-71-37 which exhibited the most acceptable characteristics was further evaluated for its reactions in simulated functional and system environments. MLO-71-37 was found to possess the most satisfactory overall properties and exhibited potential operational capability over a temperature range of -65 to 275 F. All three candidate fluids displayed a sensitivity to elastomeric materials, with specific manufacturer and compound designations required for satisfactory performance.

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SECTION I

INTRODUCTION

Tertiary phosphate esters (Fig. 1) have found wide usage as fireresistant hydraulic fluids in commercial aircraft and their ground support equipment. These materials have not been widely used in current operational military aircraft and support equipment because of incompatibility with currently used MIL-H-5606(C) hydraulic fluid, elastomers, paints and electrical insulation. Their limited high temperature capability of +225°F also prevents their use in most of the newer military aircraft. General characteristics of the tertiary phosphate esters are well known and have been covered in great detail in the literature (1)(2). A recent advance in the state-of-the-art within this area has be n the development of the so called, "Low Density Phosphate Esters". These low density phosphate esters have found wide acceptance and usage in hydraulic systems of commercial aircraft. The exact chemical composition of these low density phosphate esters is proprietary knowledge of their producers (of which there are several), however, they are probably primarily trialkyl phosphate esters. These low density phosphate esters have better low temperature properties, somewhat lower densities and are significantly lower in cost than the previously used alkyl-aryl phosphate esters. Fire resistance of the low density phosphate esters is good but somewhat lower than that of the alkyl-aryl phosphate esters. Phosphate ester hydraulic fluids are covered under Boeing Aircraft Company Material Specification BMS-311-C Hydraulic Fluid, Fire Resistant. This specification is used by industry and the commercial airlines. It covers three fluid types. Type III deals with the low density phosphate esters.

TERTIARY PHOSPHATE ESTERS

WHERE R', R'& R" ARE ARYL OR ALKYL GROUPS

FIGURE 1 STREETURE, TERTIARY PROSPUTE ESTERS

Although the low density phosphate ester hydraulic fluids are not suitable for use in most current operational military aircraft without extreme and costly retrofit or risking operational failure by accidental mixing of the phosphate esters with currently used petroleum hydraulic fluids (they are absolutely non-compatible), they are of concern to the Air Force because they are used in commercial aircraft purchased and used by the Air Force. For example, it is used in Soeing 707 (VC-137) aircraft used for transport of personnel and cargo.

Military Specification MIL-H-83306 (USAF) Hydraulic Fluid, Fire Resistant, Phosphate Ester Base, Aircraft cowers low density fire resistant bydraulic fluids for use over the temperature range of -55 to +225°F in Air Force owned and operated commercial aircraft (designed to use these materials in their hydraulic systems). This specification was issued to insure quality control and fluid performance of the low density phosphate esters used by the Air Force.

Data supplied by the various producers of the low density phosphate esters indicated their operational capabilities were considerably improved over the previously used alkyl-aryl phosphate esters. This investigation was concerned with an assessment of their potential capabilities as fire resistant aerospace hydraulic fluids for advanced system use, since there are compatible seals, paints and wiring which can be used with them in new designs. It is the purpose of this document to provide the results of the characterizations conducted on the candidate low density phosphate esters.

SECTION II

MIBIALS SELECTION

Low density fire resistant phosphate ester bydraulic fluids were obtained from the macenfacturers of the three materials being used by commercial airlines. These materials were given laboratory code numbers MLO-71-37, MLO-70-62 and MLO-70-32.

SECTION III

PHYSICAL AND CHEMICAL CHARACTERIZATION

A. Thermal Stability

Thermal stability of the three low density phosphate esters was determined by the Penn State Bomb Te.t described in MIL-H-27601 with the exception that the test temperatures were 350, 400, 450 and 500°F, rather than the specified temperature of 700°F. All three of the low density phosphate ester bydramlic fluids (MLO-71-37, MLO-70-62 and MLO-70-32) had good thermal stability at 350°F (see Table I). Thermal stability tests were run at 400, 450 and 500°F on the MLO-71-37 phosphate ester bydramlic fluid which appeared to have the best overall properties based on other physical and chemical characterization results. The "bermal stability characteristics of MLO-71-37 were unsatisfactory at 400°F, and rapidly deteriorated at 450 and 500°F (Table II). Thermal stability of the phosphate exter MLO-71-37 is estimated to be about 375°F on the basis of these tests.

TABLE I

Low Density Phosphate Esters
Thermal Stability Tests
(Per MIL-H-27601*)
6 Hours at 350°F

The second is a second and the secon

	MLO-71-37	MLO-70-62	MLO-70-32
Viscosity @ 100°F, (cs)			
Original	9.93	19.19	11.19
After Exposure	9.94	10.46	11.24
Increase %	0.1	2.6	0.4
Neutralization No. mgKOH/gm	i.		
Original	0.02	0.02	0.04
After Exposure	0.06	0.07	0.05
Increase %	0.04	0.05	0.01
Appearance After Exposure	Clear	Little	Unchanged
		Change	
Corrosion Specimen Weight			
Changes, mg/sq.cm			
M-10 Steel	0.00	0.04	0.00
Bronze	0.00	0.04	0.01
52100 Steel	0.00	0.04	0.01

^{*} Except for temperature change as noted.

TABLE II

Thermal Stability Test of MLO-71-37 (Per MIL-H-27601) (1)

6 Hours @ 350° , 400° , 450° , and 500° F

	Temperature			
Viscosity @ 100°F cs Original After Exposure Increase %	350 ^o F 9.93 9.94 0.1	400°F 9.93 10.31 3.8	450 ⁰ F 9.93 11.29 13.7	500°F 9.93 410 4.028
Neutralization No. mgKOH/g Original After Exposure Increase	0.02 0.06 0.04	0.02 8.91 8.89	0.02 19.93 19.91	0.02 76.42 76.40
Appearance After Exposure	Clear	Clear	Blue to Green, Clear	Blue to Dark Brown Dark Brown Precipitate
Change in Weight of Meta mg/sq.cm M-10 Steel Bronze 52100 Steel	-0.00 -0.00 -0.00	-0.00 -0.02 -0.00	-0.01 -0.02 (2) -2.38 (3)	+0.01 (4) -0.46 (5) -5.1 (6)

- (1) Except for temperature changes as noted.
- (2) Some area of gray discoloration.
- (3) Etched: gray discoloration.
 (4) Dark amber discoloration.
- (5) Coppertone discoloration.
- (6) Corrosion, etched, white gray discoloration.

B. Shear Stability

Shear stability of the low density phosphate ester hydraulic fluids was determined by the Sonic Shear Method as specified in MIL-H-5606(C) (Table III). Two of the phosphate ester fluids (MLO-70-62 and MLO-71-37) exhibited satisfactory shear resistance (conformed to MIL-H-5606(B)); however, the other phosphate ester fluid (MLO-70-32) had much poorer shear resistance and failed to meet MIL-H-5606(C) requirements. All three fluids however, conform to MIL-H-83306 (USAF) in all other respects.

C. Lubricity

Lubricity of the low density phosphate ester fluids was determined by the Shell Four-Ball Wear Test Method. The results of the tests, shown in Table IV, reveal that the wear characteristics of three candidate fluids are within the requirements of specifications MIL-H-5606(C) and MIL-H-83306 (USAF). Following these tests a more extensive low/temperature wear profile was conducted with fluid MLO-71-37. The results of this effort are presented in Table V. Some unusual occurrences were noted on the four-ball specimens which were run at 350°F and 40 kilogram loads. There was an unusual accumulation of a very viscous soap-like material at the edge of the wear scar, as shown in Figs. 2 and 3. Infrared spectrum and emission analysis of this material has identified it as an iron phosphate formed as a decomposition product of the organic phosphate ester fluid. Also, immediately outside of the wear scars are areas from which ball material has been removed (Figs. 4 and 5). Although the exact mechanism of this metal removal process has not been determined, the possibility exists that it results from the attack of acids formed from fluid digitated in the high temperature scar areas.

TABLE III

Shear Stability (Sonic Shear Per MIL-H-5606B)

	Reference Fluid	MLO- 71-37	MLC- 70-3:2	MLO- 70-62
Viscosity @ 130°F, cs:				
a. Before Irradiation	10.16	6.79	7.87	7.16
 b. After Irradiation 30 ml fluid for 5 min 	8.66	6.33	6.62	6.70
c. % Change	-14.76	~6.77	-15.92	-6.42
Viscosity @ -40°F, cs:				
a. Before Irradiation	385.9	414.2	319.2	303.4
b. After Irradiation30 ml fluid for 5 min	341.4	376.8	270.9	285.2
c. % Change	-11.52	~9.0	-15.12*	-6.01
Neutralization No. mgKOH/g				
a. Before Irradiation		0.02	0.04	0.02
b. After Irradiation30 ml sample for 30 min	# W W	0.22	0.34	0.24
c. Change		+0.20	+0.30	+0.22

^{*} Failed

TABLE IV

Shell Four-Ball Wear Data

(167°F, 600 rpm, 40 kg Load, 2 Hours Using 52100 Steel Balls)

MLO-Number Fluid	Average Wear Scar Diameter (mm)
MLO-70-32	0.556
MLO-70-62	0.469
MLO-71-37	0.567

TABLE V

Four-Ball Wear Test Results MLO-71-37

Avg Scar Dias in mm

SPEED: 600 rpm TIME: 1 Hour Ball Dia: 0.500 in.

Temperature ⁰ F	Material	1	Load, 4	Kilogra 10	ms 20	40
167	52100 M-10	. 21 . 17	.27 .23	. 33 . 28	. 40 . 37	. 49 . 49
200	52100 M-10					. 60 . 55
259	52100 M-10					. 64 . 56
300	52100 M-10					.71 .55
350	52100 M-10					. 73 . 55



FIGURE 2 WEAR SCAR WITH MATERIAL DEPOSIT



FIGURE 3 WEAR SCAR WITH MATERIAL DEPOSIT

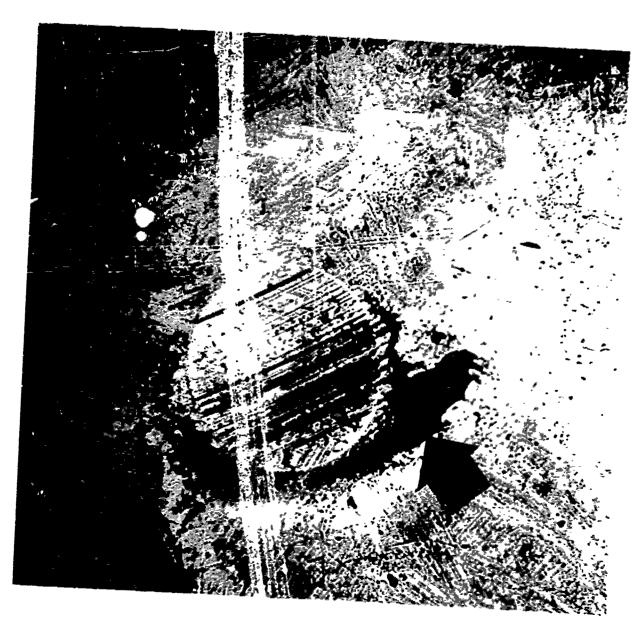


FIGURE L WEAR SCAR WITH UNUSUAL METAL REMOVAL



FIGURE 5 WEAR SCAR WITH UNUSUAL METAL REMOVAL

TABLE VI

Hydrolytic Stability Tests

168 Hours @ 225°F MIL-H-5606(B) Amendment III

(0.87% by Weight of Distilled Water) (99.13% by Weight of Oil Sample)

Tests On The Original Oil				
	MLC 0-32	MLO-71-37	MLO-70-62	
Viscosity @ 130°F, cs	7.64	6.62	6.76	
Neutralization No. mgKOH/g	3.04	0.06	0.03	
Tests On	The Hydrolyzed	Oil		
Viscosity @ 130°F, cs	8 02	7.01	6.70	
Neutralization No. mgKOH/g	9.01	0.01	9.03	
Evaporation Loss, %	1.3	2.4	1.75	
Appearance After Hydrolysis	(ilive greei	n Greenish	No change	
	No Ppt.	Blue/ No Ppt.	No Ppt.	
Increase In Viscosity, 3	5.(5.9 ⁻	0.9	
Decrease In Neutralization No. mgKOH/g	6.03	0.05	0.00	
Weight Change	Of Metals, n	ng/sq.cm		
Magnesium	+0.01 (1)	0.01	0.00	
Aluminum	0.00	0.00	0.00	
Copper	-0.04 (2)	0.01 (4)	0.02 (2)	
Cadmium	0.00 (3)	0.03	0.01 (2)	
Steel	0.00 (1)	0.00	0.00 (3)	

- Brown Spots
 Moderate Tarnish 2C
 Light Bronze and Brown Spots
 Moderate Tarnish 2A

TABLE VII

Hydrolytic Stability Tests

168 Fours @ 275°F

(0.87% by Weight of Distilled Water) (99.13% by Weight of Oil Sample)

Tests On The Original Oil

	MLO-70-62	MLO-71-37	MLO-70-32
Viscosity @ 130°F, cs	6.76	5.62	7.64
Neutralization No., mgKOH/g	0.03	0.06	0.03
Tests On	The Hydrolyzed	Oil	
Viscosity @ 130°F, cs	11.58	7.21	8.41
Neutralization No., mgKOR/g	25.29	0.01	0.09
Evaporation Loss &	5.5	1.4	3.4
Appearance After Hydrolysis	Dark Brown	Light Brown	Brown
	No Ppt	No Ppt	No Ppt
Increase In Visocisty %	7.13	8.9	10.1
Increase In Neutralization No.,	25.26	0.05	0.06
Weight Change	Of Metals m	g/sq.cm	
Magnesium	4.91(1)	0.01	÷0.02 (5)
Aluminum	9.00	0.00	0.00
Copper	32.35(2)	0.04 (4)	-0.13 (6)
Cadmium	40.97(1)	0.02	0.00 (7)
Steel	0.00	0.00	(8) 00.0

- (1) Pitted and gray discoloration.
- (2) Etched, pitted.
- (3) Pitted.
- (4) Moderate tarnish 2B.
- (5) Gray discoloration.
- (6) Moderate Tarnish 2C.
- (7) Light brown discoloration.
- (8) Light brown discoloration and spots.

TEMES TITLE

Corrosion and Oxidation Stability Tests (Per MIL-H-5606)

168 Hours At 275°F

Tests On The Original Oil

			
	MLO-70-32	MLO-70-62	MLO-71-32
Viscosity @ 130°F, cs	7.87	7.16	6.79
Neutralization No., mgKOH/g	0.0 4	0.02	0.02
Tests On	The Oxidized	Oil	
Viscosity @ 130°F, cs	8.19	9.28	7, 12
Neutralization No., mgKOH/g	0.04	6.50	0.02
Evaporation Loss &	1.3	7.1	0.96
Appearance After Oxidation	Brown	Dark Brown	Light Brown
	No Ppt		-
Increase In Visocisty %	4.1	29.6	4.9
Change in Neutralization No., mgKOH/g	0.00	6.48	0.00
Weight Ciran	ge Of Metals,	mg/sq.cm	
Magnesium	0.00	0.56 (1)	9.00
Aluminum	0.00	0.00 `´	0.00
Copper	0.01(1)	3.31 (2)	0.02 (3)
æel	0.00 `	0.00 `	0.60
Cadmium	0.01 (2)	15.70 (1)	0.02

- (1) Moderate tarnish 2C.
- (2) Light bronze discoloration.(3) Moderate tarnish 2B.

ver II

Fluid Viscosities (cs)

Temperature	MLO-71-37	MTO-19-35	MLO-70-52
-65 ⁰ F	1691	1130	1135
-40°F	414.2	319.2	303.4
100 ⁰ p	9.93	11.19	10.19
210°F	3.27	3.93	3.5

D. Hydrolytic, Stability Test

The hydrolytic stability of the three low density phosphate ester hydraulic fluids was determined by evaluating them in a modified oxidation corrosion test. The test method and procedures of MIL-H-5606(B) were used with the following modifications:

- (a) 0.87% by weight of distilled water was combined with 99.13% by weight of oil sample:
- (b) separate tests were run at 225°F for 168 hours and at 275°F for 168 hours.

All three of the low density phosphate ester hydraulic fluids displayed adequate hydrolytic stability at 225°F (see Table VI). However, at 275°F, one of the low density phosphate ester fluids (MLO-70-62) degraded severely while the other two phosphate ester fluids still gave satisfactory results (Table VII).

E. Oxidation and Corrosion Stability

The corrosion and oxidation stability of these low density phosphate ester hydraulic fluids was determined by evaluation using the Oxidation and Corrosion test specified in MIL-H-5606(B). Test temperature was 275°F.

Two of the low density phosphate ester hydraulic fluids, MLO-70-32 and MLO-71-37, gave satisfactory results while the MLO-70-62 low density phosphate ester fluid was severely degraded (Table VIII).

F. Viscosity

The viscosities of the low density phosphate ester hydraulic fluids were measured at -65, -40, 100 and 210°F (Table IX). The viscosities of all the fluids met the viscosity requirements of MIL-H-83306 (USAF). The low density phosphate esters have excellent low temperature viscosities equal to or better than other currently used aircraft hydraulic fluids.

G. Volatility

The volatility of the low density phosphate ester MLO-71-37 was determined by the Isoteniscope Method. Results are shown in Table X. A comparison is made with the volatility of MIL-H-5606(B).

H. Fire Resistance

Assessment of the fire resistance of the low density phosphate esters was not the purpose of this project; however, a few typical flammibility tests were made. Test results are shown in Table XI.

I. Elastomer Compatibility

Elastomer compatibility was investigated with ethylene propylene (ERP) "O" rings immersed in the low density phosphate ester fluids at 275°F for 72 hours. The compatibility of the low density phosphate esters with ethylene propylene elastomers varies greatly, depending upon the particular ethylene propylene elastomer used. Results range from satisfactory to disastrous (Table XII). EPR "O" ring B completely determ at d in all three of the low density phosphate ester fluids in static evaluation tests at 275°F with low density phosphate ester MLO-70-62. Manufacturer's data on EPR compound A show satisfactory compatibility with all three low density phosphate ester candidates at 275°F, as shown in Table XIII. Consequently, from these elastomer-fluid compatibility studies, O-ring packings of elastomer compound A were selected for dynamic packing tests with candidate fluid MLO-71-37.

J. Dynamic Packing Tests

Dynamic packing tests were conducted with candidate fluid MLO-71-37 and O-rings packings of EPR compound A, according to the procedures set forth in Military Specification MIL-P-25732B, paragraphs 4.6.6 and 4.6.7.1, with modifications to permit elevated temperatures and changes in fluid

TÁBLE X

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Fluid Volatility
Vapor Pressure (mm Hg)
Isoteniscope Method

		MIL-H-5606(B)
Temperature (OF)	MLO-71-37	(MLO-69-58)
100		0.2
150	0.25	1.1
200	1.10	4.7
256	3.70	15.5
300	11.30	46
350	39.0	120
400	71.0	270
450	27.7	530
500	990	1 1

TABLE XI

Fire Resistance

Flash Point (^O F) (Cleveland Open Cups) ASTM D-92	MLO-71-37 345	MLO-70-62	MLO-70.53	W''.O-76-74"
Fire Point (^O F) ASTM D-92	400	390	\$80	410
Spontaneous Ignition Temperature				
ASTM D-286 ASTM D-2155	1000+ 725	1000+ 740	1000+ 765	1000- 946
Flame Propagation Test Propagation Rate (cm/sec)	ivc Flame Advance	No Flame Advance	\$ 8 8	¢ •

* Alkyl-aryl phorphate ester hydraulic fluid type used by airlines before introduction of low density phosphate esters

TABLE ALL

Rubber Compatibility 72 Hours @ 275°F (Fluid MLO-70-62)

	EPR "O" Ring A	EPR "O" Ring 2
% Swell	17.29	125.3
Durometer Hardness		
Before Test	70	
After Test	66	
Elongation (inches)		
Before Test	1.8	Gress Elastomer
After Test	1.3	Degradation - No Evaluation Possible
Tensile Strength (psi)		
Before Test	167	
After Test	86	
	Condition of MLO-70-62	Fluid
Viscosity @ 100°F cs		
Before Test	10.19	10.19
After Test	10.44	10.99
Increase %	2.4	7.8
Neu alization No. mgKOir/g		
Before Test	0.02	0.03
After Test	0.16	0.08
Increase	0.14	0.05

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TABLE XIII

Rubber Compatibility EPR "O" Ring A 72 Hours @ 275°F

Fluid	Swell	Tensil Strength (psi)	Hardness Shore "A"	Elongation
M2.O-71-37	÷16	1886	70	236
MLO-70-52	+14	1925	70	224
MLO-70-32	+13	1829	72	226
Original "O" Ring Properties		1925	80	200

and elastomer types. The results of tests at 275, 300 and 350°F are presented in Table XIV. Although packing tests results were satisfactory at 300° F, fluid degradation at this temperature limits systems use to 275° F.

K. Hydraulic Pump Circuit

The ultimate judgment of a hydraulic fluid candidate must be made on its ability to properly function in the environment of an aircraft hydraulic system. Consequently, from its performance in the chemical and physical bench tests, low density phosphate ester candidate MLO-71-37 was selected for evaluation in the hydraulic pump circuit.

The hydraulic pump circuit, shown schematically in Fig. 6, has demonstrated the ability to simulate the physical (thermal and rechanical stresses) and chemical (materials) environment of aircraft hydraulic systems with controlled operating parameters.

The aircraft type hydraulic pump, a high temperature check valve design, was retrofitted with new elastomeric components made from EPR compound A.

Fluid evaluations were made at temperatures of 275, 300, and 350°F for periods of 50 hours, or until either the pump or fluid exhibited signs of degradation or failure. Operating conditions for these evaluations are shown in Table XV.

Operational problems were few and of little significance. The 275°F test was interrupted at 15 hours due to a power outage. This test was terminated at 32.5 hours due to fluid loss from an instrument line failure. This termination was not considered significant since there were no signs of fluid or pump degradation. The 360°F fluid evaluation was interrupted at 16.2 hours due to a pump drive control malfunction.

Fluid property changes exhibited by the low density phosphate ester fluid MLO-71-37 during the three hydraulic pump circuit tests are shown in Tables XVI, XVII and XVIII.

TABLE XIV

Dynamic Packing Test Results

FLUID: MLO-71-37
PACKING: Elastomer Compound "A"

275°F Test	Leakage (ml)	
1	3.0	All Packings Excellent
2	5.8	15 ft 11
2 3	6.5	11 11 11
4	8.0	11 11
5	8.5	Very slight nibbling on drag ring O.D.
6	Endurance	Packing failed at 78,00° cycles. Total leakage 38.0 ml at 66,000 cycles.
<u>300°</u> F		
1	6.0	All Packings Excellent
1 2 3	6.0	11 11 11
3	8.0	Very slight nibbling & scuffing on drag ring.
4	5.5	All Packings Excellent
5	6.5	11 11 11
6	Endurance	Packing failed at 57,100 cycles. Total leakage 34.0 ml at 36,000 cycles.
350°F		
1	Failed	Rod & cylinder packing
2	Failed	failed from combined rolling,
3	Failed	nibbling & cutting. Condition
Test series	s terminated	very bad.

Evaluations conducted to MIL-P-25732B with modifications for elevated temperatures & specimens.

TABLE XV

Operating Data For Hydraulic Pump Circuit MIO 71-37 at 275°, 300°, and 350°F

Nominal Fluid Temperature	$\mathbf{o_F}$	275	300	350
Duration of Experiment	hrs	32.5	50.0	13.1
Nominal Pump Speed	rpm	3750	3750	3750
Pump Flow Rate Maximum Flow Minimum Flow	gpm	8.39 0.96	8.49 1.02	8.29 1.24
Shear Cycles		5065	7925	2080
Pump Discharge Pressure Maximum Flow Minimum Flow	psig	2681 2950	2637 2992	2624 3020
Inlet Filter P at max flow Initial Final	psig	16 20	17 15	15 15
Temperatures Pump Case Pump Inlet Pump Discharge After Throttling Valve Chamber Atmosphere	o _F	253 258 272 278 88	271 281 295 302 98	315 333 345 351 90
Leakage, Shaft Seal	ml	75	42	30
Remaining Initial Fluid	%	30.4	82.9	87.4

TABLE XVI

MLO-71-37 Property History 275°F Hydraulic Pump Circuit Test

Sample	Viscosi		Flash(2) Point	Fire(2) Point	Acid(3) No.	
Time Hrs	100°F	210°F	o _F	o _F	mgKOH/gm	Insolubles
New	10.26	3.27	330	370	0.08	
0	9.73	3.12	310	380	0.10	
2	9.50	3.12	335	385	0.10	
4	9.36	3.01	325	380	0.14	
6	9.22	2.95	330	360	0.15	
8	9.17	2.93	310	380	0.17	
10	9.04	2.88	350	375	0.14	
15	8.76	2.81	330	370	0.15	
25	8.14	2.76	305	375	0.16	
32.5	9.25	2.98	335	370	0.21	None

ASTM Method D445
 ASTM Method D92
 ASTM Method D974

TABLE XVII

MLO-71-37 Property History 300°F Hydraulic Pump Circuit Test

Sample Time Hrs	Viscos cs 100°F		Flash(2) Point	Fire(2) Point o _F	Acid(3) No. mgKOH/gm	Insolubles
0	9.34	2.97	355	370	0.12	
2	9.30	2.95	340	375	0.13	
4	9.20	2.96	325	380	0.15	
6	9.23	2.90	330	380	0.16	
8	9.18	2.87	325	385	0.17	
10	8.91	2.84	345	390	0.19	
16.2	9.31	2.97	350	390	0.11	None
16.2	9.24	2.95	350	385	0.16	
25	8.63	2.88	335	380	0.32	
50	8.63	2.71	355	385	8.22	Trace

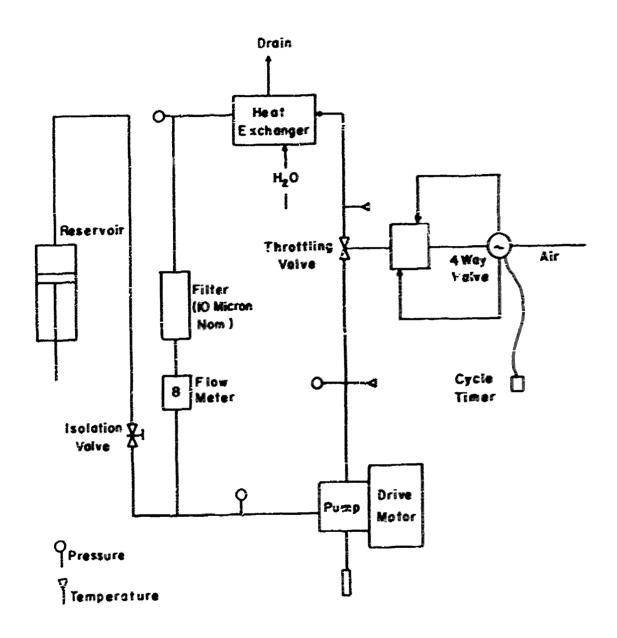
⁽¹⁾ ASTM Method D445(2) ASTM Method D92(3) ASTM Method D974

TABLE XVIII

MLO-71-37 Property History 350°F Hydraulic Pump Circuit Test

Sample Time	Viscosity(1) cs		Flash(2) Point	Fire(2) Point	Acid(3) No. mgKOH/gm	Insolubles	
Hrs	100°F	210°F	<u>°F</u>	o _F	mg von gm	<u></u>	
0	9.16	2.85	340	380	1.10		
2	9.20	2.91	330	375	10.18		
4	9.29	2.93	315	365	8.66		
6	9.45	2.95	310	365	11.76	Trace	
8	9.69	3.07	325	360	19.17	Trace	
10	10.00	3.17	345	3 50	21.88	Trace	
13.1	9.89	3, 14	325	375	16.01	Trace	

ASTM Method D445
 ASTM Method D92
 ASTM Method D974



PIGURE 6 HYDRAULIC PUMP CIRCUIT SCHEMATIC

There were no fluid property changes during the 275°F evaluation which could be considered as indicative of gross fluid degradation. During the 300°F evaluation, a sharp increase in acid rember, accompanied by a trace of insolubles, was produced at between 25 and 50 hours. These charges were viewed as signs of incipient fluid degradation. The fluid evaluation at 35FF produced signs of gross fluid degradation at 2 test bours with a sharp increase is acid number and confirmed the suspected incipient fluid breakdown during the ROPF test. A precipitate, produced during the 300°F evaluation, was separated and washed with solvents to a pure white poster. This powder was subjected to spectral analysis and identified as an iron phosphate. It was hypothesized that the strong acids produced by the fluid degradation reacted with the from containing system components to produce the iron phosphate. The color of the fluid samples from the 3500% eralization changed from the original clear medium blue to blue-green to yellor-oraspr-

The stainless steel element filters contained a small amount of light grey polationess residue and some fine copper colored particles after the 275°F evaluation. After the 350°F test, the pump discharge filter and the disc elements contained a considerable quantity of copper colored material from the pistom slippers and the pump thrust bearing. Also, the filter elements retained a thin but apparently porous film of pelatizous or grease-like material. From the residue on the four-ball specimens tested at 150 $^{\circ}$ F and 40 kilograms and the material in the filter, it appears that the thermal degradation of Mir-Ti-37 produces a celatinous by-product.

The aircraft type bydraulic pump was disassems, of for a pre-test inspection and after each of the fluid evaluations. There was no evidence of pump degradation from either eismai examination at clanure in the weights of the pump friction elements following the ITS and Roof? Sheid evaluations

However, after the 350°F test, pump elements exhibited signs of gross wear from lubrication failure to the point that a pump overhaul would be required to restore it to operating condition. The primary areas of wear were the copper alloy piston heads and thrust bearing, which exhibited degradation severe enough to xender them unserviceable. (The degree of wear, as indicated by element weight loss for various pump parts, is presented in Table XIX.) Although the pump was in excellent condition, the incipient fluid degradation at the end of the 300°F test limits the use of the candidate fluid to a maximum temperature of 275°F.

TABLE XIX

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Hydraulic Pump Friction Element Weight Changes Low Density Hydraulic Fluid MLO-71-37

Total	2260	0836	0250	4687
Change After 350 ⁰ F Test	1896	0824	0103	4624
Change After 300 ⁰ F Test	0079	0007	0003	0043
Change After 275 ⁰ F Test	0285	0005	0143	0020
	Pump Piston Assemblies*	*	* <u>.</u>	6)
	Piston	Pump Collars*	Pump Sleeves*	Nutation Plate
	Pump	Pump	dund 34	Nutatio

All weights in grams.

* Average of seven elements.

SECTION IV

CONCLUSIONS

The three low density phosphate ester fluids evaluated were suitable for use under Military Specification MIL-H-83306 (USAF) Hydraulic Fluid, Fire Resistant, Phosphate Ester Base; and Boeing Aircraft Company Material Specification EMS-311-C Hydraulic Fluid, Fire Resistant. These specifications cover operational ranges of -65 to +225°F and -65 to +225/250°F, respectively. There are, however, significant differences in the physical and chemical characteristics of the candidate fluids. The low density phosphate ester MLO-70-32, exhibited greater susceptibility to shearing action than the other two fluids (MLO-70-32 failed the shear resistant requirement of MIL-H-5606(B)). The low density phosphate ester MLO-70-62 was hydrolytically and oxidatively unstable and corrosive to metals at 275°F, whereas the other two fluids, MLO-70-32 and MLO-71-37, were satisfactory.

The low density phosphate ester with the best overall properties, as determined by the chemical tests, exhibited very good characteristics in simulated hydraulic pump and actuator systems to 275°F. This temperature limit was established by incipient fluid degradation at 300°F in the hydraulic pump circuit at 50 test hours.

The low density phosphate ester, MLO-71-37, which had shown the best overall properties in this evaluation, exhibited potential operational capability over the -65 to 275°F temperature range. Extreme care is required in the selection of elastomeric materials for use with the low density phosphate ester fluids. The use of properly specified elastomers must be complied with for satisfactory results.

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- Hatton, R. E., "Introduction to Hydraulic Fluids", Reinhold Publishing Corp., New York City, 1st ed., 1962, pp. 190-211.

Security Classification DOCUMENT CONTROL DATA - R&D Security classification of title, body of abstract and indexing ampotation must be intered when the overall report is classified) 2. REPORT SECURITY CLASSIFICATION JAIGINATING ACTIVITY Competers sucher Air Force Materials Laboratory Unclassified Wright-Patterson AFR, Ohio 45433 25 SROUP 3 REPORT TITLE Chemical Physical and Mechanical Properties of low Density Phosphate Ester Hydraulic Fluids DESCRIPTIVE MOTES (Type of report and inclusive dates) March 1973 through June 1972 \$ AJTHOR'S, 'Last name first name imital) F. Brooks and H. Schwenker TO TOTAL NO OF PAGES 76 NO OF REFS S REPORT CATE April 1973 36 SE ORIGINATOR'S REPORT NUMBER(S) SA COMINACT OR SHANT NO. AFMI_TR-73-78 & PROJECT NO 7340 OTHER REPORT NO(3) (Any other number of the mount) 4 734008 is available, by Libitation workers Distribution limited to U. S. Government agencies only (test and evaluation) March 1973. Other requests for this document must be referred to the Air Force Materials Laboratory, Nonmetallic Materials Division, Lubricants &Tribology Branch, AFML/NBT Wright-Patterson Air Force Base. Ohio 45433 IT SUPPLEMENTARY NOTES 12 SPONSORING WILITARY ACTIVITY Air Force Materials Laboratory Wright-Patterson AFR, Ohio 45433 TOARTESA EL Three low density phosphate ester fluid candidates, MLO-70-32, MID-70-62 and MID-71-37, were characterized as to their physical and chemical properties. MLO-71-37 which exhibited the most acceptable characteristics was further evaluated for its reactions in simulated functional and system environments. MLO-71-37 was found to possess the most satisfactory overall properties and exhibited potential operational capability over a temperature range of -65 to 275 F. All three candidate fluids displayed a sensitivity to elastomeric materials with specific manufacturer and compound designations required for satisfactory performance.

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